

C L A I M S

1. A crystalline, hydrated form of the sodium salt of 3-pyridyl-1-hydroxyethylidene-1,1-bisphosphonic acid, wherein the form contains from 6.4 up to 22 weight % of sodium, based on the anhydrous substance, and 15 up to 23 weight % of crystalline water if the sodium content is lower than 7.5 weight %, or 4.5 up to 18 weight % if the sodium content is higher than 7.5 weight %.
- 5 2. The crystalline form according to claim 1, which is pentahydrate of the monosodium salt of 3-pyridyl-1-hydroxyethylidene-1,1-bisphosphonic acid, wherein said form contains 20 up to 23 weight % of water built in the crystal lattice and 5.5 up to 7.5 % of sodium.
- 10 3. The crystalline form according to claim 2 wherein said form contains 22.8 weight % of water built in the crystal lattice and 6.4 up to 6.7 % of sodium.
- 15 4. The crystalline form according to claims 2 or 3 wherein said form shows a powder X-ray diffraction pattern with interplanar distances d approximately 16.3; 13.0; 9.1 and 4.9 Å.
- 20 5. The crystalline form according to claims 2 or 3 wherein said form shows the infrared spectrum with bands 1169; 1060; 1046 and 891 cm⁻¹.
6. The crystalline form according to claims 2 or 3 thermogravimetric analysis of which shows a plateau at temperature of about 173 °C.
- 25 7. The crystalline form according to claims 2 or 3 the ³¹P CP-MAS NMR spectrum of which shows signals 13.7 and 20.0 ppm.
- 30 8. The crystalline form according to claim 1, which is trihydrate of the trisodium salt of 3-pyridyl-1-hydroxyethylidene-1,1-bisphosphonic acid, wherein said form contains

19 up to 21 weight % of sodium and 12 up to 14 weight % of water in the crystal lattice.

9. The crystalline form according to claim 8 wherein said form shows the infrared spectrum with bands approximately 1114; 1085; 956 ; 616 and 544 cm⁻¹.

5 10. The crystalline form according to claim 1, which is monohydrate of the disodium salt of 3-pyridyl-1-hydroxyethylidene-1,1-bisphosphonic acid, wherein said form contains 13 up to 15 weight % of sodium, based on the anhydrous substance, and 4.5 up to 6.5
10 weight % of water in the crystal lattice.

11. The crystalline form according to claim 10 wherein said form shows the infrared spectrum with bands approximately 1183; 1158; 1071 and 1042 cm⁻¹.

15 12. A method of manufacture of the crystalline form according to any of the foregoing claims *characterized in* that an aqueous solution of the sodium salt of 3-pyridyl-1-hydroxyethylidene-1,1-bisphosphonic acid heated to 50 up to 80 °C is incorporated into an organic solvent.

20 13. The method according to claim 12 *characterized in* that the organic solvent is selected from the group of simple alcohols from the C₁ to C₅ series, especially 2-propanol.

25 14. The method according to any of claims 1 through 11 *characterized in* that seeding crystals of the respective hydrate of the sodium salt of 3-pyridyl-1-hydroxyethylidene-1,1-bisphosphonate are introduced into the solution of the sodium salt of 3-pyridyl-1-hydroxyethylidene-1,1-bisphosphonic acid and the solution is slowly cooled.

30 15. The method according to claim 14 *characterized in* that crystallization is performed from a solution of the sodium salt in a mixture of water and a water-miscible organic substance.

16. A pharmaceutical composition designed for treating diseases associated with a bone-resorption disorder *characterized in* that it contains as the active agent a hydrate of the sodium salt of 3-pyridyl-1-hydroxyethylidene-1,1-bisphosphonic acid according to any of claims 1 through 11 and at least one auxiliary substance.